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Certification of the Standard Reference Material 1473a, A Low Density Polyethylene Resin

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Certain commercial materials and equipment are identified in this paper in order to specify adequately the experimental procedure. In no case does such identification imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply necessarily the best available for the purpose.

ABSTRACT

The melt flow rate of Standard Reference Material (SRM) 1473a, a polyethylene resin, was determined to be 1.17 g/10 min at 190 °C under a load of 2.16 kg using the ASTM Method D 1238-90b. The average results from 66 determinations on samples with a standard deviation of a single measurement of 0.015 g/10 min. A small but measurable drift from the first timed extrudate to the third timed extrudate was observed.

1. Introduction

Melt flow rate as determined by ASTM D 1238-90b is widely used in polymer technology as a product specification since this value, which includes a statement of the load and temperature under which it is obtained, gives an indication of the processing properties of the polymer[1-4]. The value of melt flow rate is expressed as the mass of polymer melt pushed from the heated cylinder of the extrusion plastometer through its precision bore orifice by its piston in a period of time, the standard units of the value being grams per ten minutes (g/10 min) as prescribed by ASTM D 1238-90b.

This is a report on the melt flow rate certification of SRM 1473a, a polyethylene resin, with ASTM D 1238 melt index conditions, 190 °C temperature and 2.16 kg load. SRM 1473a is the designated successor to SRM 1473 the characterization of which was described in an earlier report[5]. SRM 1473 and SRM 1473a are taken from different lots of the same type of resin from the same supplier, Quantum Chemical Corp., USI Division, Cincinnati, OH. A small but reproducible difference in melt index between the two lots of resin was observed.

2. Experimental Procedure

2.1 Sampling and Randomization of Charge Sequence of SRM 1473a

Material of the SRM 1473a came in one 50 pound bag of pellets. The material was selected as a result of a search for a polyethylene with a melt index nearly equal to the melt index of an earlier SRM, SRM 1476. The supplier of this resin identifies it as a low density polyethylene synthesized in an autoclave environment which characteristically generates a branched product. The supplier estimates the density to be 0.918 g/cm^3. The Standard Reference Materials Program (SRMP) blended the material and divided the pellets into 315 units of 60 grams each. Eleven of these units were chosen by stratified random selection for homogeneity and certification studies. Six charges were taken for extrusion from each of the randomly chosen bottled samples during the course of the melt flow rate experiments. In preparation for the melt flow rate determinations, the charges to be extruded were identified by ordinal numbers. Six such charge numbers were assigned to each identified sample. The sequence of numbered charges taken from the bottled samples for extrusion was randomized according to a procedure described by Natrella[6], and applying the Rand tables[7].

2.2 Instrument Calibration and Alignment

2.2.1 Temperature Indication

The temperature of the extrusion plastometer cylinder was indicated by a mercury column thermometer of the form described in paragraph 5.7 of the ASTM method. Calibration of the temperature indication is traceable to the Thermometry Group of the NIST Process Measurements Division. An iron-constantan thermocouple was calibrated by correlating its output voltage (emf) with the scale readings of an ASTM 68C thermometer at 10 points from 185 °C to 194 °C, in a constant temperature oil bath. The ASTM 68C thermometer had been calibrated at the ice point and at 190 °C in the Thermometry Group of the NIST Process Measurements Division by comparison with a platinum resistance thermometer. The temperature indication by the scale of the cylinder thermometer in the extrusion plastometer was calibrated by correlating the thermometer scale readings with the temperature indicated by the emf from the calibrated thermocouple with its hot junction stationed in a column of polyethylene melt in the bore, as described in paragraph 5.5.2 of the ASTM method. Thermal conductivity between cylinder and thermometer was enhanced by adding Wood's metal to the thermometer well in the cylinder. The uncertainty in the final temperature indication may be regarded as equal to the nominal limit of resolution on the scales of the ASTM 68C thermometer, and of the thermometer in the extrusion plastometer, 0.1 °C. The effect of a 0.1 deg uncertainty in temperature on the melt index of SRM 1473a is described in the subsequent section on uncertainty analysis.

2.2.2 Metering of Plastometer Components

The geometric dimensions of the cylinder, piston assembly, and dies were found to comply with the specifications described in the ASTM method. The length and outer diameter of the piston feet and dies were determined by a micrometer. The bore of the dies was tested by a pair of gauges with diameters equal to the minimum and maximum tolerance limits specified in the ASTM method.

The diameter of the cylinder bore was determined by a Brown and Sharpe model 599-281 Intrimik inner diameter (ID) micrometer. The ID of the bore was measured at the bottom end (micrometer head resting on a die at the bottom), and at levels from 15 cm down from the top end, up to 2 cm from the top end, in 1 cm intervals. The resulting measurements varied from 0.9543 cm to 0.9550 cm, in compliance with the tolerance of this specification described in paragraph 5.2 of the ASTM method.

The apparent mass of the nominal 2060 g load was determined in the Mass Group of the NIST Automated Production

Technology Division, and found to be 2059.8 g. A set of new detachable piston feet was used to conduct the extrusions, the foot being changed on the piston for each extrusion. The assembled piston varied in apparent mass from 100.027 g with the lightest foot attached, to 100.071 with the heaviest foot attached. Thus the calculated combined apparent mass of piston and load varied from 2159.8_3 g with the lightest foot attached to the piston, to 2159.8_7 g with the heaviest foot attached to the piston, both limits well within the $\pm$0.5% tolerance described in paragraph 5.4.4 of the ASTM method.

2.2.3 Alignment of Plastometer

The cylindrical axis of the bore was aligned with the gravity vector by the following plumb-line procedure.

First, a die was selected as the "target" die and stationed on the structural baseplate of the extrusion plastometer directly below the cylinder. Its position on the plane of the baseplate was adjusted to have the axis of its bore coincide with the projection from the axis of the cylinder bore onto that plane. This was accomplished by viewing the target die through the bores of two "sighting" dies, one stationed at its operational position in the bottom end of the cylinder bore and the other stationed at the top end of the cylinder bore. The position of the target die on the plane of the baseplate was adjusted until it appeared centered in the view from above the cylinder through the sighting dies in the cylinder bore.

Next a plumb-bob was suspended by a plumb-line from the axis of a die supported in the top end of the cylinder bore, with the pointer of the suspended bob extending down inside the bore of the target die. The leveling screws were adjusted until the pointer of the plumb-bob appeared to be centered inside the bore of the target die.

The deviation of the plumb bob pointer was observed to be less than 1 mm from the point which would indicate ideally vertical orientation of the cylinder bore, at the end of a pendulum length of 41 cm. Consequently, this procedure is considered to obtain alignment of the cylindrical axis of the cylinder bore with the gravity vector, with a calculated uncertainty of (1 mm/41 cm). Thus, the maximum uncertainty in terms of possible angle of displacement of the bore axis from the gravity vector is estimated as arcsin(1mm/41cm)=0.14°. It is estimated that such a maximum displacement in angle from the gravity vector would diminish the force of the loaded piston to a level of its total weight multiplied by cos 0.14°, or (2.16 kg)(0.999997) in the present case. This factor would thus diminish the effective weight of the loaded piston by a decrement of 3E-4%, much less

than the ±0.5% tolerance in combined weight of piston and load described in paragraph 5.4.4 of the ASTM method.

The initial bore alignment was conducted at ambient temperature in preparation for the characterization. The alignment was also occasionally tested, during the course of the characterization extrusions while the cylinder was hot, with a circular level which can be mounted atop the piston rod mounted in the bore, as described in the bore alignment section of the ASTM method.

3.0 Melt Flow Rate of SRM 1473a

The melt flow rates of SRM 1473a samples were determined by procedure A described in Section 9 of ASTM Method D-1238-90b[8]. Standard test condition 190/2.16 was used. Thus the flow rate was determined at 190.0 ±0.1 °C using a load of 2.16 kg. The flow rate of the melt was measured by a manually operated extrusion plastometer obtained from the Tinius Olsen Testing Machine Co.

A 3.2 g charge of pellets was used for each extrusion. The end of the 6 minute preheat period was marked as the beginning of timed test extrusion by making the initial extrudate cut at 6 minutes and discarding the preheat segment. It was also observed that the 4 mm start section of the piston had always entered the top of the guide collar part way at the moment of the initial cut to begin collecting timed test extrudate. Three timed test extrudate segments were cut at 3 minute intervals thereafter. After the third timed test extrudate segment had been cut, the remaining melt in the cylinder was purged and discarded. Following ASTM D1238 only the first timed extrudate was used in the final data analysis for certification of SRM 1473a.

The piston and bore were cleaned free of the polymer at the end of each extrusion. Many dies and piston feet were obtained from the Tinius Olsen Testing Machine Co. During a given day a different die and piston foot was used for each separate charge. The dies and piston feet were then cleaned in preparation for the next days run. Tools of brass and copper, considerably less hard than steel, were applied in the cleaning process. The use of steel tools was avoided in order to prevent changing dimensions of instrument components due to cleaning wear.

Two operators were used in the operation of the equipment. Each operator made measurements on eight or nine extrusions (charges) on alternate days.

4.0 Data Analysis on SRM 1473a

Data from 66 charges were analyzed for the 2.16 kg load following the ASTM Method D1238-90b. The average melt flow rate of SRM 1473a was found to be 1.17 g/10 min with a standard deviation of a single measurement of 0.015 g/10 min. with 65 degrees of freedom. This standard deviation includes bottle to bottle, charge to charge, operator to operator and day to day variability. The standard deviation of the mean was calculated to be 0.0019 g/10 min. Our estimates of systematic uncertainties in the measurement will be discussed in the following sections.

5.0 Uncertainty Estimates of Melt Flow Rate Data

The measurement uncertainties encountered in the process of determining the melt index were determined or estimated in compliance with the recently documented NIST policy governing the reporting of uncertainties in measurement[10]. Uncertainties cited in this report are considered equivalent to those corresponding to a 95% level of confidence. The uncertainty due to instrument variability among the results from a large population of laboratories was derived from expected precision limits tabulated in the ASTM method description[8].

5.1 Repeatability and Sampling Uncertainties

In this section we discuss the uncertainty in terms of variations of the measured melt flow rate arising from sampling and bottling differences.

The standard deviation of a single measurement was 0.015 g/10 min for 66 melt flow rate determinations in a range from 1.12_8 g/10 min to 1.20_1 g/10 min. The standard uncertainty of our results, due to overall experimental extrusion repeatability limits, is taken as the standard deviation of the mean of all the melt index determinations, u = 0.00185 g/10 min. The relative standard uncertainty, given as 0.00185/1.17 = 0.158%, is listed in Table 1.

5.2 Charge to Charge Variability Within a Bottle

As described in Section 2.1, 11 bottles were selected for measurement from the original bottling. Six charges from each bottle were measured so a study of charge to charge variation within an individual bottle was easily performed. The mean charge to charge standard deviation of the point of the melt flow value from charges removed from an individual bottle was 0.014 g/10 min

with a range from 0.004 to 0.022 g/10 min. This data is not reported separately in Table 1, since this uncertainty is included in the relative standard uncertainty reported in line 1 of Table 1.

5.3 Bottle to Bottle Variability

The bottle to bottle variability was estimated from the population of all charges taken from all 11 bottles. The mean value of the melt flow rate for any bottle was found to lie within two standard deviations of the mean melt flow rate for all the charges. The mean of any bottle was not found to be significantly different at the 95% confidence level from any other mean in the group. This data is not reported separately in Table 1 since this uncertainty is included in the overall standard uncertainty reported in line 1 of Table 1.

5.4 Day to Day Variability

Eight to nine melt flow rate experiments could be conducted in a single day. The day to day variability was small compared to the charge to charge variability. This data is not reported separately in Table 1, since this uncertainty is included in the overall standard uncertainty reported in line 1 of Table 1.

5.5 Operator to Operator Variability

Eight to nine melt flow rate determinations could be conducted in a single day. On alternate days different operators were used. The mean melt flow rate determined by operator 1 was 1.168 g/10 min with a standard deviation of a single measurement of 0.0167 g/10 min. The mean melt flow rate determined by operator 2 was 1.165 g/10 min with a standard deviation of a single measurement of 0.0133 g/10 min. Thus the operator to operator variability appears to be negligible in comparison with the charge to charge variability. This uncertainty is not reported separately in Table 1 since it is included in the overall standard uncertainty reported in line 1 of Table 1.

5.6 Systematic Uncertainties

Obtaining a systematic uncertainty analysis of the melt flow rate is a difficult matter since the melt flow rate is not a fundamental property of the material and there is no simple relationship describing its estimation. Nonetheless we shall make an effort in this section to estimate the uncertainties from their possible contributing sources, and their contribution to the combined uncertainty to be computed.

5.6.1 Instrument Variability

As noted before, the estimates of our own repeatability are in line 1 of Table 1. These data reflect the repeatability of our own experiments and do not reflect any instrument-to-instrument variation since we had only one instrument.

However, the results in Table 5 in ASTM D1238-90b provide a means of estimating the uncertainty among the results from a large population of instruments and operators applying procedure A. The average melt flow rate for SRM 1473a, 1.17 g/10 min, is closely comparable with the average melt flow rate, 2.04 g/10 min, for another polyethylene under the same conditions, listed in Table 5 of ASTM D 1238-90b, and averaged from the results reported from nine laboratories. Their tables of precision limits lists repeatability standard deviations, S_r, and reproducibility standard deviations, S_R, in addition to other related statistical parameters, all defined in ASTM Standard E 691-92, "Standard Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method"[9]. The standard deviation of the mean of an array of average melt index results from a large population of laboratories can be derived from the S_r and S_R given in the precision tables of ASTM D 1238-90b by applying their relationship with the more fundamental standard deviation of the mean described in ASTM E 691-92. Thus a standard deviation of the mean, s_x = 0.077 g/10 min, was derived for the average melt index 2.04 g/10 min from its corresponding S_r and S_R listed in Table 5 of ASTM D 1238-90b. Expressed in terms of percent, s_x = 3.77%, this standard deviation of the mean is applied as an estimate of the standard uncertainty for the currently reported average melt index, 1.17 g/10 min, due to instrument variability in the results from a large population of laboratories. This standard uncertainty due to instrument variability is also listed in Table 1 of this report. Predicated on statistical analysis of measurements from nine laboratories in a study coordinated by ASTM Committee D-20, this is a Type A uncertainty in the nomenclature described in NIST Technical Note 1297.

5.6.2 Measurement Uncertainties

An effort is made to estimate the intrinsic uncertainty in the measurement. We do this by considering the uncertainties in the measured quantities (mass and time) as well as the uncertainties in the controlled quantities (temperature). The melt flow rate, FR, is given by

$$FR = Mass/Time$$

Thus the relative uncertainty in the melt flow rate, u_{FR}/FR, is then obtained as the root-sum-of-squares of the uncertainties in

the systematic physical factors upon which the melt flow rate depends[10],

$$(u_{FR}/FR)^2 = (u_m/m)^2 + (u_t/t)^2 + (c_T*u_T/FR)^2,$$

where the fractional uncertainty in weight of extrudate is u_m/m, u_t/t is the fractional uncertainty in timing of the extrudate cut and the u_T/FR term arises from the uncertainty in the temperature control and in the calibration of temperature indication. The factor c_T is the sensitivity coefficient, $c_T=(\partial FR/\partial T)$. The causes of these uncertainties are discussed in the next few paragraphs. These with the other uncertainties are given in Table 1 as well as an estimate of the combined standard uncertainty resulting from all sources.

5.6.2.1 Weighing Uncertainty

The extrudate segments were weighed on a balance with 0.01 mg resolution and an estimated uncertainty of 0.05 mg. Replicate weighings of the segments always agreed to within ± 0.05 mg. Paragraph 9.9 of ASTM Method D 1238-90b instructs the experimenter to "weigh the extrudate to the nearest 1 mg when cool."

The extrudate segments were routinely weighed within one hour after having been cut, in compliance with the instruction in paragraph 9.9 of the ASTM method. Considering the hydrophobic character of polyethylene it would not be anticipated that the extrudate would accumulate moisture beyond the initial cooling stage prior to being weighed. On a few occasions during the characterization of another polyolefin, extrudate segments, which had been weighed at the end of a day, were weighed again on the following day without detecting any statistically valid change of weight within the groups. All individual changes, either positive or negative, were much smaller than 0.1 mg.

The extrudate weight was about 350 mg. With an estimated overall weighing uncertainty of 0.15 mg from the above sources, the relative uncertainty

$$u_m/m = 0.04\%.$$

Since this weighing uncertainty appears negligible in comparison with larger uncertainties from other sources, we take the weighing uncertainty to be zero.

5.6.2.2 Timing Uncertainty

The 3 min interval (t = 180 s) between extrudate cuts for SRM 1473a was measured with a battery powered stopwatch having a

0.01 s resolution in time indication and an uncertainty of less than 0.05 s. Thus the extrudate cut was assumed to be timed to better than 0.1 s. Consequently, we take 0.1 s as a practical estimate of the timing uncertainty. Hence, the relative uncertainty in time interval may be expressed

$$u_t/t = \pm 0.1 \text{ s}/180 \text{ s} = 0.06\%$$

We take the timing uncertainty as zero, since it appears negligible in comparison with larger uncertainties from other sources.

5.6.2.3 Temperature Uncertainty

As described in section 4.2.1, the extrusion cylinder temperature was indicated by a mercury column thermometer of the form described in paragraph 5.7 of the ASTM method. The uncertainty of the thermometer is certified to be within the tolerances of the ASTM method, by comparison to standards traceable to NIST. The uncertainty in the temperature indication calibration is less than 0.1 °C.

Paragraph 5.7 in the ASTM method acknowledges that the temperature in the thermometer well may not necessarily be the temperature of the polymer melt at the calibration point in the bore. This is due to the steady state heat transfer gradients in the plastometer cylinder. Thus, the thermal profile of an undisturbed column of polyethylene melt was scanned along the cylindrical axis of the cylinder bore while the temperature was maintained at 190.0 °C at the calibration point in the melt column. This experiment was conducted in another extrusion plastometer during an earlier determination of the melt flow rate of SRM 1475. The column of melt was held stationary by plugging the flow. The temperature in the stationary melt column was measured with a thermocouple hot junction stationed at different heights above the top surface of the die, along the cylindrical axis of the bore. Throughout the experiment the reading of the mercury column thermometer remained at 190.0 ± 0.1 °C. The results are listed in Table 2.

Inspection of the tabulated results indicates that the departure of melt temperature from the indicated cylinder temperature is within ± 0.1 °C at any location in the melt column from 12 mm above the die upward. There is a 0.7 deg drop in temperature between the 12 mm and 1 mm levels above the die. This temperature drop is probably at least partially erased by the downward flow of melt during an extrusion. Since we have no other measurements on this profile, we may suppose another column may have a different profile. We take the 0.7 deg temperature drop determined at the bottom end of the melt column adjacent to

the die, observed in Table 2, as an estimate of the temperature uncertainty due to possible thermal gradients in the melt column. This uncertainty is combined with the nominal uncertainty in temperature indication, 0.1 deg, by root-sum-of-squares, and the result rounded up to 0.8 deg, as an estimate of maximum expected standard uncertainty in temperature, u_T, due to temperature indication and to possible gradients in the thermal profile of the melt column.

The effect of small variations in temperature on the apparent melt index was determined during the characterization of the original SRM 1473 reported earlier[5]. Thus, the effect of temperature variation on melt flow rate was determined by conducting a set of five extrusions at 188.4 °C, and another set of five extrusions at 191.5 °C. The two sets of extrusions at the different temperatures were conducted with charges of polyethylene resin all taken from the same bottle of SRM 1473.

The resulting average melt flow rates from the first timed extrudate segments of those extrusions, and from that at 190 °C, are taken from Table 4 of the earlier report and listed in Table 3 of this report. Linear regression analysis of the tabulated melt flow rate versus temperature provided the slope

$$(dFR/dT) = (0.040 \text{ g/10 min}\cdot)/°C$$

taken as the quantitative expression for the temperature dependence of the melt flow rate of SRM 1473 at temperatures near 190 °C. A plot of melt flow rate versus temperature is seen in Figure 1.

Since SRM 1473 and SRM 1473a are different lots of production of the same type of polyethylene from the same manufacturer, this (dFR/dT) for SRM 1473 is also taken as the sensitivity coefficient, c_T, of the flow rate to variation in temperature of the melt for SRM 1473a.

Considering the standard uncertainty in temperature, u_T = 0.8 °C, this result affords an estimate of ± 0.032 g/10 min for the uncertainty in melt flow rate of SRM 1473a due to uncertainty in temperature. The relative uncertainty in melt flow rate due to uncertainty in temperature is then 0.032/1.17, or 2.7%.

5.7 Comparison with Earlier Melt Flow Measurements on SRM 1473

As discussed in sections 1 and 2.1, the material for SRM 1473a is a low density polyethylene resin obtained from a single batch from the manufacturer. The original bottling of SRM 1473 was obtained from the same manufacturer a number of years

earlier. It was of a different batch and is thus expected to not have exactly the same melt flow. As reported in NISTIR 4627, SRM 1473 has a melt flow rate of 1.29 g/10 min compared to the value of 1.17 g/10 min under the same conditions reported here for SRM 1473a.

5.8 Combined Standard Uncertainty

The combined standard uncertainty[10] for the melt flow rate, u_c, is obtained as the root-sum-of-squares of component uncertainties from all sources. The combined standard uncertainty computed thus from the sources discussed in the preceding paragraphs is u_c = 4.63%, neglecting the uncertainties due to weighing and timing. When the uncertainties in weighing and timing were included in the computation, the resulting u_c was 4.64%. The difference between the two values is negligible in the subsequent calculation of the expanded uncertainty. The combined standard uncertainty is listed in line 6 of Table 1.

5.9 Expanded Uncertainty

The reproducibilities, I_R, listed in the precision tables of ASTM D 1238-90b are a form of 95% confidence interval estimate[11]. With the object of presenting an uncertainty consistent with the reproducibilities tabulated in the ASTM method, the combined standard uncertainty from Table 1 of this report is expanded to the form of a 95% confidence interval estimate. This is accomplished by applying a coverage factor of 2 to the combined standard uncertainty[12] to obtain an expanded uncertainty, $U=2u_c$. The resulting expanded uncertainty is 9.3% for the melt index of SRM 1473a. This 95% confidence interval estimate is closely comparable with the reproducibility, equivalent to 11%, listed in Table 5 of ASTM D 1238-90b for their polyethylene with melt index 2.04 g/10 min.

6.0 Conclusions of Melt Flow Rate Study

The melt flow rate of SRM 1473a was found to be 1.17 g/10 min, with a standard deviation of an average single measurement of 0.015 g/10 min, and a standard deviation of the mean of 0.0019 g/10 min. The combined standard uncertainty is 4.64%, and the expanded uncertainty is U = 9.3%, equivalent to 0.11 g/10 min in melt flow rate units.

Acknowledgment

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11. Paragraph 13.1.6 in reference 8.

12. Paragraph 6.2 in reference 10.

Table 1

Estimates of Uncertainties in Melt Flow Rate, FR, of SRM 1473a Polyethylene Under Condition 190/2.16

Source of Uncertainty	u_i % of FR	Type[c]
1. Uncertainty due to repeatability of experiment	0.16%	A
2. Uncertainty due to instrument variability as estimated from I_r and I_R factors in precision tables in ASTM D 1238-90b	3.77%	A
3. $u_m/m*100$ (weighing uncertainty)	0.04%	B
4. $u_t/t*100$ (timing uncertainty)	0.06%	B
5. $(c_T^a*u_T/FR)*100$ (temp. uncertainty)	2.7%	B
Combined standard uncertainty, u_c[b]	4.64%	
Expanded uncertainty, $U = 2u_c$[12]	9.3%	

a. Sensitivity coefficient, $c_T = dFR/dT$, for variation of flow rate in response to small changes in melt temperature.

b. The combined standard uncertainty is computed by root-sum-of-squares of the component uncertainties[10].

c. Type of uncertainty[10]:
Type A uncertainties are evaluated by statistical methods.
Type B uncertainties are evaluated by other means.

Table 2

Variation of Temperature with Height in Undisturbed Melt in Cylinder Bore

Height Above Die, mm	Melt Temp. °C
48	190.09
36	189.93
24	189.97
12	189.94
1	189.23

Table 3

Temperature Dependence of the Melt Flow Rate (FR) of SRM 1473 in the Vicinity of 190 °C Under 2.16 Kg Load (Reference 5)

Temp. °C	FR-T/2.16, g/10 min.
188.4	1.227
190	1.287
191.5	1.352

(dFR/dT) = 0.040 g/10 min. per degree

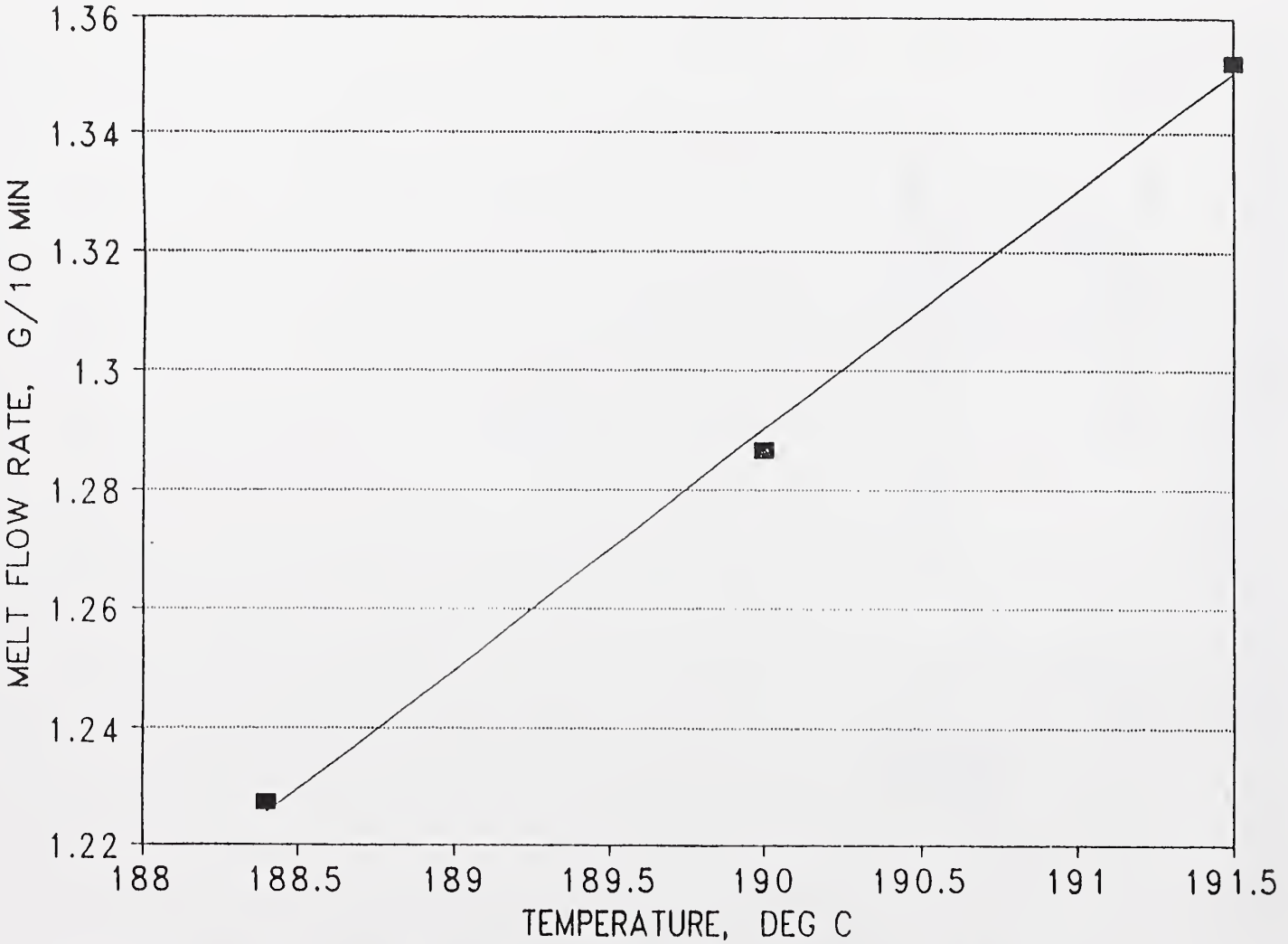

Figure 1. Temperature Dependence of Melt Flow Rate of SRM 1473 Under 2.16 kg. Load Near 190°C

National Institute of Standards & Technology

Certificate

Standard Reference Material® 1473a

Low Density Polyethylene Resin

This Standard Reference Material (SRM) is intended primarily for use in calibration and performance evaluation of instruments used in polymer technology for the determination of the melt flow rate. The SRM is supplied as white pellets of polyethylene in a 60 g unit.

This material is certified for melt flow rate, FR-190/2.16, using ASTM Method D 1238-90b, Standard Test Method for Flow Rates of Thermoplastics by Extrusion Plastometer [1] Standard test condition 190/2.16. That is, the flow rate of the melt was determined at 190.0 °C ± 0.1 °C using a load of 2.16 kg by procedure A of the ASTM method, using a manually operated extrusion plastometer. Under these conditions [2], the certified melt flow rate for this material is as follows:

Melt Flow Rate (FR) = 1.17 g/10 min ± 0.11 g/10 min

Uncertainty: The uncertainty is the numerical value of an expanded uncertainty $U = ku_c$, with U determined from a combined standard uncertainty, u_c, and coverage factor $k = 2$ [3] at a level of confidence of 95 %. The standard deviation for an average single measurement is 0.015 g/10 min, with 65 degrees of freedom [2].

Source of Material: The supplier for this material was Quantum Chemical Corp., USI Division, Cincinnati, OH.

NOTICE AND WARNING TO USERS

Expiration of Certification: This certification will be valid for five years from the date of shipment from NIST.

Storage: SRM 1473a should be stored in the tightly closed, original bottle under normal laboratory conditions.

The technical coordination leading to certification of this material was provided by B.M. Fanconi of the NIST Polymers Division. Technical measurement and data interpretation were provided by J.R. Maurey, W.R. Blair, and C.M. Guttman of the NIST Polymers Division.

The technical and support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by J.C. Colbert.

Gaithersburg, MD 20899
August 31, 1995

Thomas E. Gills, Chief
Standard Reference Materials Program

Table 1. Estimate of Uncertainties [3] in Melt Flow Rate (FR) of SRM 1473a Polyethylene under ASTM Standard Test condition 190/2.16

Source of Uncertainty (u_i)	Type[c]	u_i (as % of FR)
1. Uncertainty due to repeatability of experiment	A	0.16 %
2. Uncertainty due to instrument variability as estimated from I_r and I_R factors in precision tables in ASTM D 1238-90b	A	3.77 %
3. $u_m/m \cdot 100$ (weighing uncertainty)	B	0.04 %
4. $u_t/t \cdot 100$ (timing uncertainty)	B	0.06 %
5. $(c_T^a \cdot u_T/FR) \cdot 100$ (temp. uncertainty)	B	2.7 %
Combined standard uncertainty, u_c[b]		4.64 %
Expanded uncertainty, $U = 2u_c$		9.3 %

[a]Sensitivity coefficient, $c_T = d(FR)/dT$, for variation of flow rate in response to small changes in melt temperature.
[b]The combined standard uncertainty is computed by root-sum-of-squares of the component uncertainties.
[c]Type of uncertainty - Type A uncertainties are evaluated by statistical methods: Type B uncertainties are evaluated by other means.

REFERENCES

[1] D 1238-90b, 1994 Annual Book of ASTM Standards, Vol. 08.01, pp 272-280, (1994).

[2] Maurey, J.R., Blair, W.R., and Guttman, C.M.: "Certification of the Standard Reference Material 1473a, A Low Density Polyethylene Resin", NISTIR 5639, (1995).

[3] Taylor, B.N. and Kuyatt, C.E.; "Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results", NIST Tech. Note 1297, September, (1994).